The first SANS instruments utilizing long flight paths, long wavelength neutrons from a reactor cold source and position sensitive detectors were developed over 40 years ago. Small-angle neutron scattering instruments should really be called low-Q instruments. Q is the scattering variable which is expressed in terms of the neutron wavelength λ and low scattering angle θ as $Q = 2\pi\theta/\lambda$. Low Q can be realized either through the use of small angles or long wavelengths (or both). In order to obtain small angles, good collimation and good resolution area detectors are needed. Good collimation is achieved through the use of long neutron flight paths before and after the sample. SANS instruments on continuous neutron sources use velocity selectors to select a slice of the (often cold) neutron spectrum while time-of-flight SANS instruments use a wide slice of the spectral distribution with careful timing between the source chopper and the detector to separate out the various wavelength frames. In this last case (TOF instruments) the maximum length of an instrument is determined by the pulse frequency so as to avoid frame overlap (whereby the fastest neutrons of one pulse would catch up with the slowest neutrons of the previous pulse).

1. CONTINUOUS SANS INSTRUMENT COMPONENTS

A brief description of the main components of reactor-based SANS instruments follows. This description covers the main features found on the NG3 30 m SANS instrument at the NIST Center for Neutron Research (Hammouda et al, 1993; Glinka et al, 1998).

-- Cold neutrons are transported through total internal reflection at glancing angles inside neutron guides. These transmit neutrons from the cold source to the entrance of scattering instruments with little loss (1 % per meter). Neutron guides are coated with natural Ni or Ni-58 which has a wider critical angle for reflection. This critical angle increases linearly with neutron wavelength thereby allowing more cold neutrons to reach the SANS instrument. Note that supermirrors (characterized by even higher critical angles) are not used due to the tight collimation requirement of SANS instruments; neutrons that experience too many reflections never make it through the tight SANS collimation.

-- Beam filters (for example Be for neutrons and Bi for gammas) are used to clean up the beam from unwanted epithermal neutrons and gamma rays. Gammas are stopped by high-Z materials such as Bi. Be transmits neutrons with wavelengths > 4 Å. Note that if a curved guide is used, no crystal filter is needed because there is no direct line-of-sight from the reactor source (no gammas in the beam). Curved guides transmit only wavelengths above a cutoff value (no epithermal neutrons in the beam). Typical crystal filter thickness is between 15 cm and 20 cm. For better efficiency, filters are cooled down to liquid nitrogen temperature (77 K = -196 ᵒC).

-- Optical filters are devices that stir a neutron beam away from the direct line-of-sight and replace crystal filters. They consist of tapered neutron guides that transmit only

neutrons that are reflected. They have the advantage of gains in flux over crystal filters at long wavelengths.
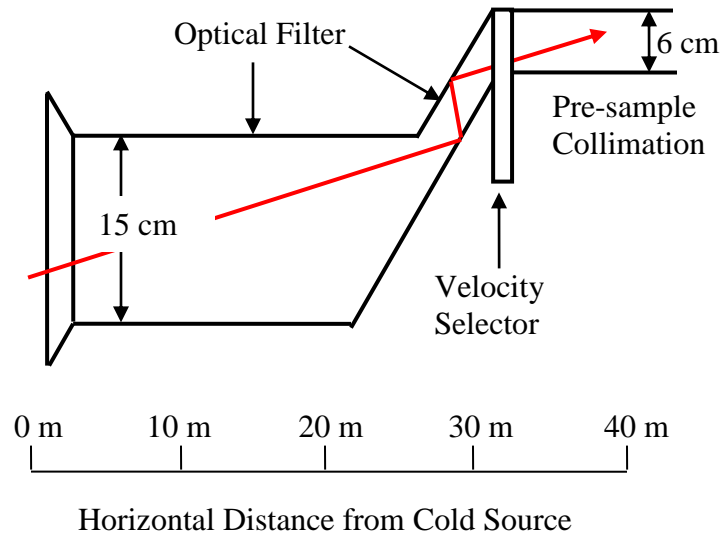

Figure 1: Schematic side view representation of an optical filter used on the NG3 SANS instrument at the NIST CNR facility. The reflecting guide surfaces are made out of Ni and Ni-58. Since there was no room horizontally, the neutron beam is steered vertically. This figure is not to scale.

-- A velocity selector yields a monochromatic beam (with wavelengths λ between 4 Å and 20 Å and wavelength spreads $\Delta\lambda/\lambda$ between 10 % and 30 %). Some SANS instruments that need sharp wavelength resolution use crystal monochromators (with wide mosaic spreads to give $\Delta\lambda/\lambda < 10$ %) instead. Since $\Delta\lambda/\lambda$ is constant, the neutron spectrum transmitted by the velocity selector falls off as $1/\lambda^4$ (instead of the $1/\lambda^5$ coming from the moderator produced Maxwellian distribution).
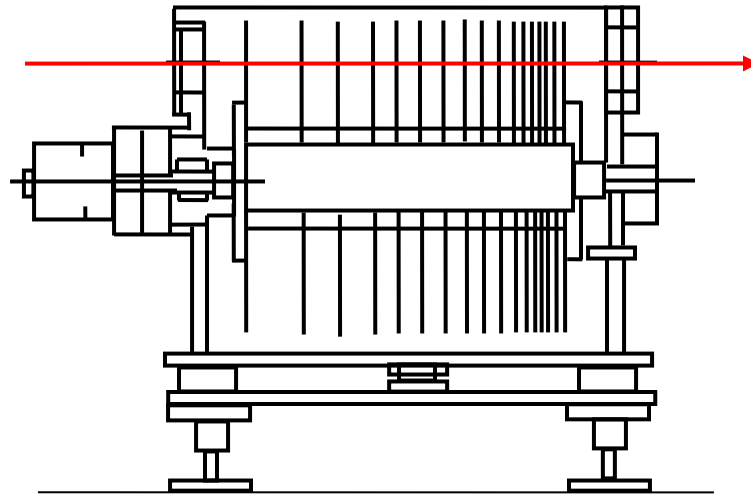
Figure 2: Schematic representation of a multi-disk velocity selector. A white neutron spectrum is incident from the left and a monochromated beam is transmitted toward the right. Changing the rotation speed controls the neutron wavelength. Tilting the selector horizontally changes the wavelength spread. Magnetic coupling is used to drive the selector rotation to high rotation speed. Temperature and vibration sensors insure reliable operation.

-- The collimation usually consists of a set of circular (source and sample) apertures that converge onto the detector. An evacuated pre-sample flight path contains the beam collimation system. Typical adjustable flight path distances are from 1 m to 20 m depending on resolution and intensity considerations. Inside the pre-sample flight path, more neutron guides (with reflecting inner surfaces) are included in parallel with the collimation system for easy insertion into the beam. This allows a useful way to adjust the desired flux on sample along with the desired instrumental resolution by varying the effective source-to-sample distance. A normal configuration consists of a certain number of guides inserted into the neutron beam, followed by a source aperture right after, then a series of empty beam positions up to the sample aperture located inside the sample chamber.
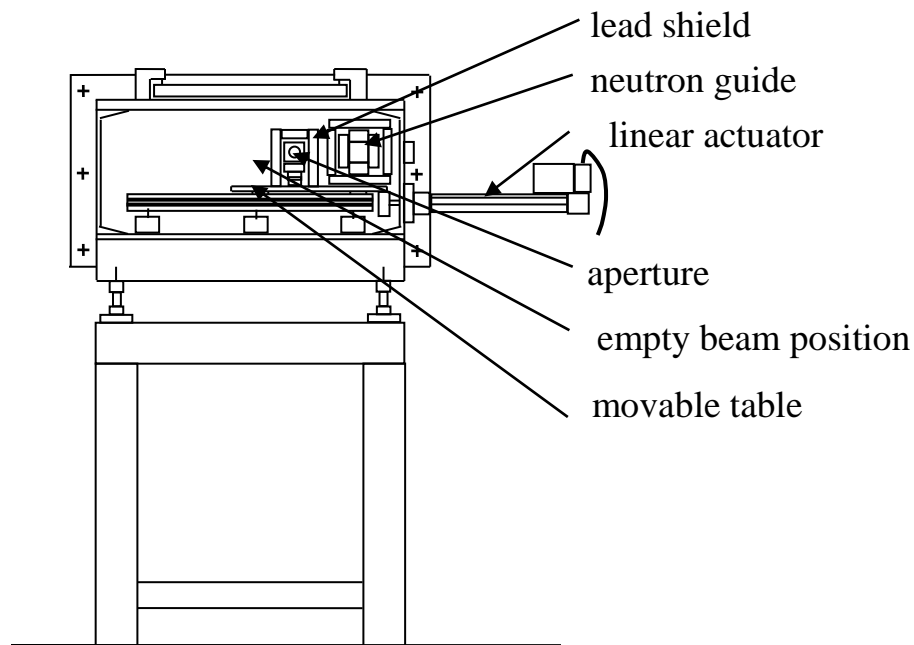
Figure 3: Cross section of a pre-sample flight path box showing an aperture, a neutron guide and an empty beam position that can be inserted in the beam using an actuator and a movable translation table.

-- A sample chamber usually contains a translation frame that can hold many samples (measured in sequence). Heating and cooling of samples (-10 ^oC to 200 ^oC) as well as other sample environments (cryostats, electromagnets, ovens, shearing devices, etc) are often accommodated. The oversized sample environments are mounted on a 22" diameter Huber sample table instead. This sample table can be rotated around a vertical axis and translated in and out of the neutron beam. This translation is useful for moving the in-situ rheometer (for example) from the radial position to the tangential position. All of these motions are computer controlled.
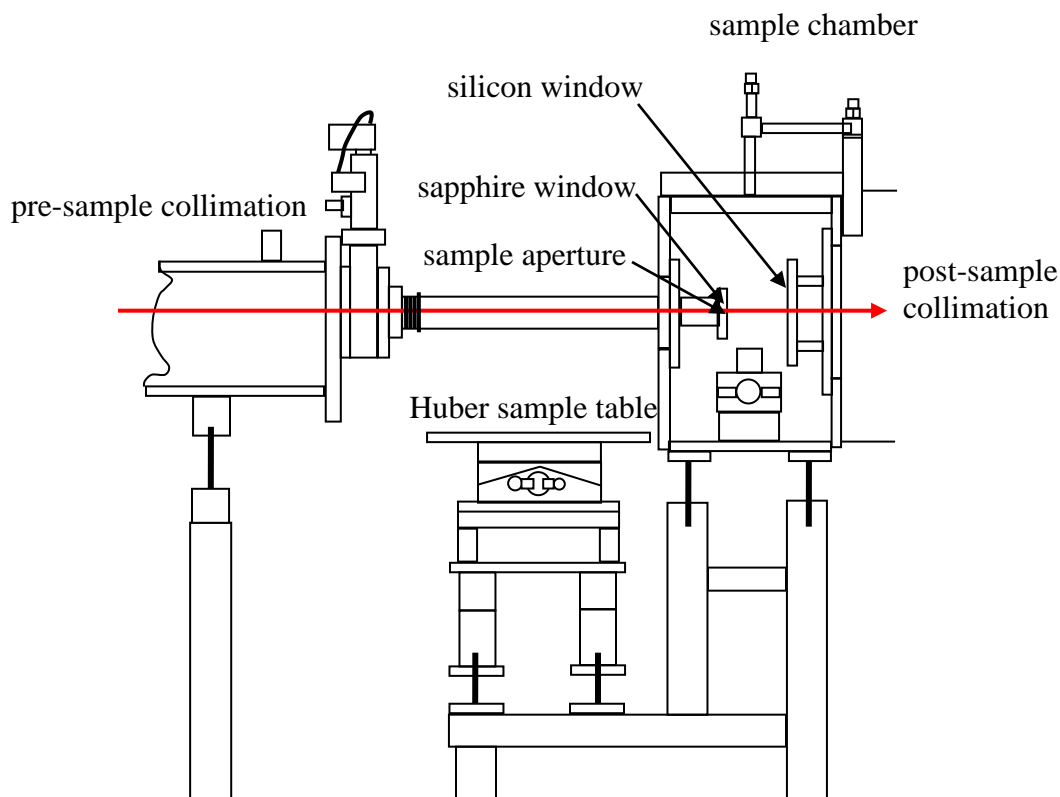
Figure 4: Schematic drawing of the sample area showing the sample chamber on the right and a sample table for oversized environments on the left. Two gate valves are used to isolate the evacuated areas (pre-sample and post-sample flight path) when pumping down or evacuating the sample chamber. This softens the shock on the brittle neutron windows during such rapid pressure surges.

-- A set of three main neutron windows are used: (1) One at the entrance to the pre-sample flight path. This window is before the source aperture and can be made out of quartz. (2) A second window just before the sample. This window is between the two defining apertures and could therefore produce unwanted diffuse scattering. It is often made out of sapphire (more expensive than quartz but with better neutron transmission). Sapphire is transparent thereby allowing a laser beam (parallel to the neutron beam) through for rapid sample holder alignment. The laser itself is installed on one of the collimation boxes and produces a beam that gets reflected (90 $^{\circ}$) by a silicon mirror placed at 45 $^{\circ}$ from the beam axis. The silicon wafer reflects the laser beam but is transparent to neutrons. (3) A wide silicon window is used at the entrance to the scattering vessel (just after the sample). Silicon has the best neutron transmission and is the best window material when optical transparency is not required. These windows are between 3 mm and 6 mm thick.

-- Precise alignment of sample blocks with respect to the sample aperture is performed using a "neutron camera". A double exposure picture is taken with and without the sample aperture. A neutron camera is a regular flat camera outfitted with a scintillation plate (using material such as Li-6).

-- The post sample flight path is usually an evacuated cylindrical tube (to avoid scattering from nitrogen in air) that permits the translation of an area detector along rails in order to change the sample-to-detector distance. The vacuum level is kept at less than 100 mT. In order to evacuate such a large volume, a large capacity vacuum pump and a roots blower are used.

-- The area detector is often a gas detector with 0.5 cm to 1 cm resolution and typically 128*128 cells. The detection electronics chain starts with preamplifiers on the back of the detector and comprises amplifiers, coincidence and timing units, plus encoding modules and a means of histogramming the data and mapping them onto computer memory. In order to avoid extensive use of vacuum feedthroughs, high count rate area detector design incorporates most electronics modules (amplification, coincidence, encoding, etc) inside an electronics chamber located on the back of the detector. In this design, flexible hoses are, however, needed to ventilate the electronics and to carry the high voltage and powering cables in and the encoded signal out.
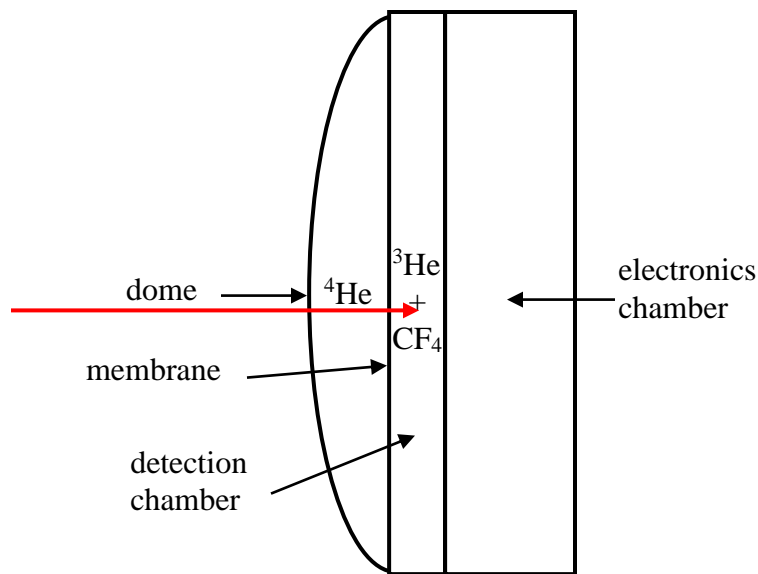
Figure 5: Schematic representation of a neutron area detector.

-- Detector protection is performed in two ways: (1) through an analog monitoring of the total count rate and (2) through software monitoring of each detector cell count rate. If either the total detector count rate or a preset number of cells overflow, the data acquisition system pauses, the next attenuator is moved in and data acquisition is restarted. Typical presets are 50,000 cps for the total detector count rate and 100 cps for a maximum of 10 cells.

-- A set of beam stops is used to prevent the unattenuated main beam from reaching the detector and therefore damaging it due to overexposure. Use of glass seeded with Li-6 as

neutron absorber avoids the gamma-ray background obtained with Cd, B or Gd containing materials. For easy alignment, motion of the beam stops should be independent of that of the area detector.

-- Between the velocity selector and the pre-sample collimation system, a low-efficiency fission chamber detector is used to monitor the neutron beam during data acquisition.

-- Just before the pre-sample collimation flight path a set of calibrated attenuators are used to attenuate the neutron beam. This system consists of a slab of plexiglass milled stepwise so as to provide attenuators of varying thickness. The insertion of this attenuator set is computer controlled. For example, if the area detector count rate is above a preset ceiling, the thinnest attenuator is moved into the beam by the data acquisition software. If this does not attenuate the beam enough, the next attenuator in thickness is moved in, etc. Another option for an attenuator system would be to use neutron absorbing material (such as cadmium) plates with holes milled into them. The density of holes would determine the attenuation factor.

-- Gamma radiation produced by neutron capture in various neutron absorbing materials (Cd, Gd, B) is stopped using high-Z shielding materials (Fe, Pb, concrete). Shields surround the velocity selector (especially the front disk that produces most of the dose) and beam defining apertures. The scattering vessel is also shielded in order to minimize background radiation from reaching the detector.

-- The pre-sample and post-sample flight paths are often made out of non magnetic metals (like aluminum or non-magnetic steel) in order to allow the use of polarized neutrons.

-- A neutron polarizer consists of a Fe/Si coating on 1 mm thick silicon plates aligned to form a V inside a copper-coated neutron guide. This polarizing cavity is 1.2 m long and polarizes a $4*5$ cm^2 neutron beam for a wavelength between 5 Å and 15 Å. Immediately following the polarizing cavity is a flat coil π spin flipper for reversing the direction of polarization. Permanent magnets maintain a 500 gauss vertical field to magnetize the supermirror coating and a 50 gauss field from the supermirror to the sample area to maintain neutron polarization.

-- In order to avoid diffuse scattering from the beam defining apertures, these are tapered (5 ° taper angle is enough) and made out of material like boron nitrite or lithiated glass with the smaller inner edge made out of cadmium. This keeps the beam sharp and any emitted gamma radiation to a minimum.

-- Data acquisition is computer controlled within menu-driven screen management environments and on-line imaging of the data is usually available. Encoded 2D data are received from the area detector electronics, binned into histogramming memories, then regularly displayed and saved. The data acquisition software interface also controls the various peripheral functions such as controlling the various motors, the sample heating/cooling protocols, and handshaking with the various other stand-alone sample

environments (shear cells or rheometers, pressure cells, etc).

-- There are many figures of merit used to judge the performance of SANS instruments. These include: instrumental resolution, minimum scattering variable (Q_{min}), flux on sample, dynamic Q range and background level.
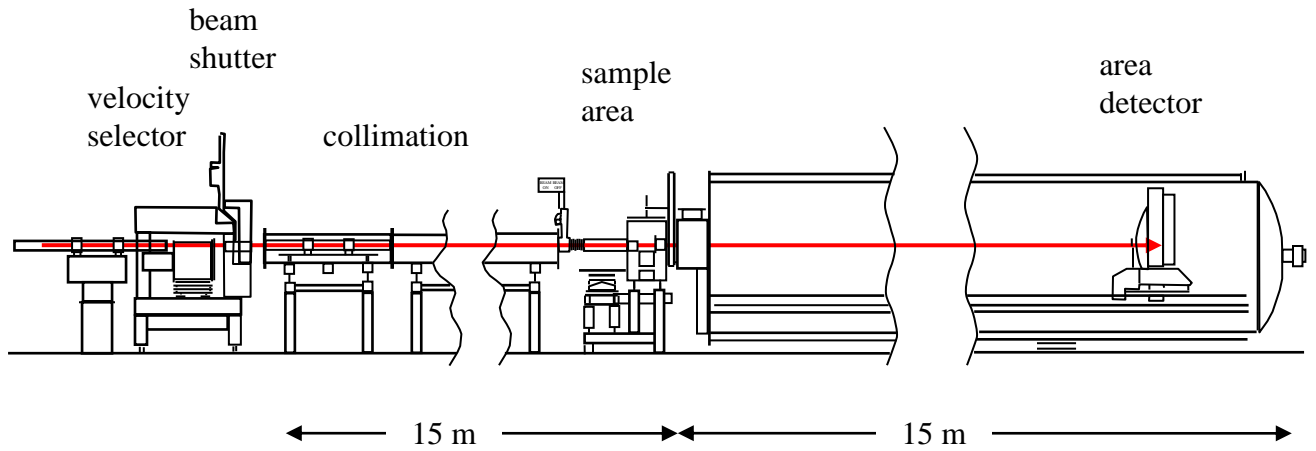

Figure 6: Schematics of a 30m SANS instrument at NIST.

Figure 7: Photograph of the NG3 30 m SANS instrument. The picture was taken from the bridge walkway just before the velocity selector shield.

Table 1: 30 m NIST-SANS Instruments Characteristics.

Source:	neutron guide (NG3), 6 * 6 cm^2
Monochromator:	mechanical velocity selector with variable speed and pitch
Wavelength Range:	variable from 5 Å to 20 Å
Wavelength Resol.:	10 to 30 % for $\Delta\lambda/\lambda$ (FWHM)
Source-to-Sample Dist.:	3.5 to 15 m in 1.5 m steps via insertion of neutron guides
Sample-to-Detector Dist.:	1.3 to 13.2 m continuously variable for NG3
Collimation:	circular pinhole collimation
Sample Size:	0.5 to 2.5 cm diameter
Q-range:	0.001 to 0.6 Å^{-1}
Size Regime:	10 to 6000 Å
Detector:	64 * 64 cm^2 He-3 position-sensitive ORDELA type proportional counter (0.508 * 0.508 cm^2 spatial resolution)

Table 2: Short list of ancillary equipment used on SANS.

Ancillary Equipment:	- Automatic multi-specimen sample changer with temperature control from -10 to 200 ^{o}C. - Electromagnet (0 to 9 Tesla). - Couette flow shearing cell, plate/plate shear cell, in-situ rheometers. - Cryostats and closed cycle refrigerators (1 K to 300 K). - Oven for in-situ use (300 K to 1800 K). - Pressure cell (0 to 1*10^8 Pa, 25 ^{o}C to 160 ^{o}C).

2. TIME-OF-FLIGHT SANS INSTRUMENT COMPONENTS

In order to avoid frame overlap, time-of-flight SANS instruments tend to be shorter at typical pulsed sources. TOF SANS instruments comprise some of the main features described above (collimation, sample chamber, flight paths, area detector, etc) as well as some specific features described here:

-- A source chopper is used to define the starting neutron pulse.

-- The area detector is synchronized to the source chopper so that a number of

wavelength frames (for example 128) are recorded for each pulse. No monochromator is necessary with the time-of-flight method.

-- A supermirror bender can be used to remove short wavelengths and let the instrument get out of the direct line of sight from the source. Note that curved guides have a cutoff wavelength below which neutrons are not transmitted. This bender replaces the crystal filter.

-- High wavelengths (say above 14 Å) have to be eliminated in order to avoid frame overlap. This can be done by gating the detector or through the use of frame overlap mirrors. Reflecting mirrors are set at a slight angle (1 ^{o}) from the beam direction so as to reflect only long wavelength neutrons (note that the reflection critical angle varies linearly with wavelength).

-- Prompt gamma rays emitted during the spallation reaction are eliminated by paralyzing the detection system for the first microsecond after each pulse.

Because of the wide wavelength range used in time-of-flight instruments, materials that display a Bragg cutoff (such as sapphire windows) cannot be used. Data reduction becomes more complex with time-of-light instruments since most corrections (transmission, monitor normalization, detector efficiency, linearity, uniformity, etc) become wavelength dependent. Time-of-flight instruments have the advantage, on the other hand, of measuring a wide Q range at once. Also the large number of wavelength frames can be kept separated therefore yielding very high wavelength resolution ($\Delta\lambda/\lambda <$ 1 %) which is useful for highly ordered scattering structures (characterized by sharp peaks).

3. SAMPLE ENVIRONMENTS

Typical sample thickness for SANS measurements is of order of 1 mm for hydrogenated samples and 2 mm for deuterated samples. Liquid samples (polymer solutions, microemulsions) are often contained in quartz or demountable cells into which syringes can be inserted. Solid polymer samples are usually melt-pressed above their softening temperature, then confined in special cells between quartz windows.

Flexibility of design for some instruments allows the use of typical size samples under temperature control or bulky sample environments. Temperature is easily varied between ambient temperature and 200 ^{o}C using heating cartridges or between -10 ^{o}C and room temperature using a circulating bath. Other sample environment equipment such as low-temperature cryostats (4 to 350 K) and electromagnets (1-10 Teslas) are sometime made available to users. Various shear cells (Couette, plate-and-plate, in-situ rheometers, etc) help probe "soft" materials at the molecular level in order to better understand their rheology. Pressure cells are also finding wide use for investigations of compressibility effects on the thermodynamics of phase separation as well as on structure and morphology.

4. SANS MEASUREMENTS

SANS measurements using cold neutrons take from a few minutes to a few hours depending on the scattering sample, the neutron source and the instrument configuration used. The process starts by sample preparation, loading into cells and measurement of the sample thickness.

A reasonable instrument configuration is chosen at first by setting a low wavelength (5 to 6 Å) and varying the sample-to-detector distance so as to optimize the desired Q-range. If the maximum available sample-to-detector distance of that instrument is reached, wavelength is then increased. Choice of the source-to-sample distance, wavelength spread, and aperture sizes are dictated by the desired instrumental resolution (sharp scattering features require good resolution) and flux on sample. Scattered intensity is controlled by many factors that have to be optimized. Transmission measurements are required as well. In order to avoid complicated multiple scattering corrections, sample transmissions are kept high (> 60 %). Many experiments require a wide Q range covering two orders of magnitude (from $Q = 0.003$ Å^{-1} to $Q = 0.3$ Å^{-1}). This range is obtained over two instrument configurations. In order to improve counting statistics, a third configuration is often used. The use of focusing lenses lowers the minimum Q down to slightly below $Q = 0.001$ Å^{-1}.

A complete set of data involves measurements from the sample, from an incoherent (usually nondeuterated) scatterer that yields a flat (Q-independent) signal, from the empty cell and blocked beam and from a calibrated (absolute standard) sample. The beam flux measurement method (measurement of the direct beam transmission) can be used to replace the absolute standard measurement.

SANS data are corrected, rescaled to give a macroscopic cross section (units of cm^{-1}) then averaged (circularly for isotropic scattering or sector-wise for anisotropic scattering). Reduced data are finally plotted using standard linear plots (Guinier, Zimm, Kratky, etc) in order to extract qualitative trends for sample characteristics (radius of gyration, correlation length, persistence length, etc) or fitted to models for more detailed data analysis.

5. SANS INSTRUMENTS IN THE WORLD

Since the first SANS instrument went into operation at the Institut Laue Langevin (Grenoble, France) in the mid-1970s, many more SANS instruments have been built. Every neutron scattering facility has at least one such instrument. The SANS technique has managed to keep a steady growth and a high level of user subscription. A web site keeps a SANS instruments directory in the world (http://www.ill.fr/lss/SANS_WD/sansdir.html).

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QUESTIONS

1. Why are small-angle neutron scattering instruments bigger than small-angle x-ray scattering instruments?
2. Why are crystal monochromators not used (instead of velocity selectors) in SANS instruments?
3. Could one perform SANS measurements without using an area detector?
4. What is the useful range of cold neutron wavelengths?
5. When is it necessary to use wide wavelength spread $\Delta\lambda/\lambda$?
6. Find out how a velocity selector works?
7. How does a He-3 area detector work?
8. What is the cost of building a SANS instrument?
9. Name some materials used for neutron windows.
10. Do cold neutrons destroy samples?
11. Why are time-of-flight SANS instruments short?

ANSWERS

1. Neutron fluxes are lower than x-ray fluxes. SANS samples are made bigger than SAXS samples in order to enhance the neutron current on sample. Bigger samples require larger flight paths in order to cover the same Q range.
2. Crystal monochromators are characterized by narrower wavelength spreads than velocity selectors and therefore lower neutron currents. Moreover, the use of a crystal monochromator would require the pivoting of the entire SANS instrument around the monochromator axis in order to change the neutron wavelength because they operate in reflection geometry. Velocity selectors operate in transmission geometry.
3. SANS measurements can be performed using an end-window or a 1D position-sensitive detector. Count rate would however be prohibitively low.
4. Cold neutron wavelengths range from 4 Å to 20 Å. The range used is effectively from 5 Å to 12 Å. Longer wavelengths are characterized by low fluxes.
5. SANS uses wide wavelength spread in order to increase the neutron current on sample.

6. Velocity selectors rotate at a specific speed for every neutron wavelength. The helical selector slot lets neutrons of the right speed through. Those that are either too slow or too fast are absorbed and never get transmitted.

7. He-3 absorbs a neutron to give two charged particles: a proton (H-1) and a triton (H-3). These two charges create an electron detection cloud that drifts towards the anode (at high voltage) and therefore get sensed by the cathodes.

8. A SANS instrument costs as much as its level of sophistication. A deluxe model costs over $ 1 million.

9. Neutron windows have to be very transparent to neutrons. Silicon is the most transparent but is opaque to visible light. Sapphire is less transparent to neutrons, very transparent to visible light but rather expensive. Quartz is like sapphire but less expensive. In practice, sapphire is used for windows before the sample. They can transmit neutrons as well as let a laser beam through for sample alignment purposes. After the sample, silicon windows are used.

10. Cold neutrons do not destroy most samples. Unlike x-rays they do not heat them up. Samples containing elements that can be activated by neutrons (such as Fe for example) have to be handled differently. Most SANS samples (polymers, complex fluids and biology) contain organic molecules that do not get activated (C, H, D, O, N, etc).

11. Time-of flight SANS instruments are short in order to avoid frame overlap (when the fast neutrons of one pulse catch up with the slow neutrons of the previous pulse).